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EFFECT OF LOW-TEMPERATURE OXIDATION ON THE HYDROGEN IN COAL AND THE CHANGE IN WEIGHT OF COAL ON DRYING

BY

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EFFECT OF LOW-TEMPERATURE OXIDATION ON THE HYDROGEN IN COAL AND THE CHANGE IN WEIGHT OF COAL ON DRYING.

By S. H. KATZ and H. C. PORTER.

INTRODUCTION.

Bituminous coal when exposed to the air at ordinary temperatures undergoes various changes that reduce its heating value. These changes affect the coal substance both chemically and physically. The molecular changes during the alteration of coal are unknown, but the changes among the different elements of the coal substance have been the subject of considerable experiment. In order to gain information regarding the possible changes of the hydrogen of the coal substance during the alteration of coal by the air, the work described in this paper was undertaken.

At ordinary temperatures oxygen combines with coal by an addition reaction and for the most part remains chemically combined with the coal substance. As a result carbon, in the form of carbon dioxide and carbon monoxide, is evolved from the coal.[a] The amount of monoxide is much smaller than that of dioxide, and together they remove from the coal a small quantity of oxygen as compared with that added during the same time.

Hydrogen makes up a portion of the coal substance which has been less investigated as regards changes during low temperature oxidation of coal.

Freshly mined coal is generally preferred for gas making or other industrial purposes. For these purposes, any loss or changes in the chemical combinations of hydrogen would affect the value of the coal. In the standard method of analyzing coal,[b] a sample is air dried before analysis, so that there is some change through oxidation. Hence there is the question as to the effect of the oxidation on the hydrogen of the coal and its bearing on the determination of water and hydrogen.

[a] Mahler, P., De l'action de l'air sur la houille: Compt. rend., t. 150, 1910, pp. 1521-1523.

[b] Stanton, F. M., and Fieldner, A. C., Methods of analyzing coal and coke: Tech. Paper 8, Bureau of Mines, 1913, 42 pp.

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RESULTS OF THE INVESTIGATION.

Nearly all of the recorded work of previous investigators of the oxidation of coal as related to the hydrogen of the coal substance was at the temperature of boiling water and above.[a] Water was invariably produced in readily determined quantities. Three different investigators have studied the subject with regard to changes at ordinary temperatures. The results obtained by these investigators vary widely—from the statement that "quite a large amount of water is produced in oxidation" to those of the authors who can find no water produced by their method of experiment. The third investigator found that in some cases very small amounts of water were produced and in others none whatever. The authors of this paper conclude that at ordinary temperatures coal undergoing oxidation produces no water. The authors have determined, too, that the change in weight of the coal is less than that of the water removed from the coal when drying takes place in an inert atmosphere. Others[b] have noted this discrepancy. A possible explanation is that the discrepancy results from the absorption of gas by the coal on drying.

PREVIOUS INVESTIGATIONS.

Porter and Ovitz[c] have shown that hydrogen escapes from coal in the form of methane when the coal is exposed after mining. Later experiments by Katz[d] show that this gas is absorbed by the coal, so that the manner of absorption is physical rather than chemical, and that hydrogen should be differentiated from that present as part of the coal substance. Consequently, this change need not be considered here.

Taffanel[e] exposed freshly made coal dusts to mine air. Analyses showed that the percentages of hydrogen, nitrogen, and volatile matter changed very little when computed on an ash-free basis. The oxygen increased and the carbon decreased. There was no evidence of changes in the absolute amount of hydrogen in the coal.

In a further study of changes in natural dusts, as they occur in the mines, Taffanel[f] says: "The carbon and hydrogen contents are diminished by the alteration; one must particularly see in this diminution the percentage compensation of the increases of the

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[a] Porter, H. C., and Ralston, O. C., A study of the oxidation of coal: Tech. Paper 65, Bureau of Mines, 1914, 30 pp. Richters, E., Ueber eine eigenthümliche Veränderung welche die Steinkohle beim erhitzen erleidet: Dingl. poly. Jour., vol. 190, 1868, pp. 398–401.

[b] Hillebrand, W. F., and Badger, W. L., Errors in the determination of moisture in coal: Original communications, Eighth Internat. Cong. Appl. Chem., vol. 10, 1912, p. 187.

[c] Porter, H. C., and Ovitz, F. K., The escape of gas from coal: Tech. Paper 2, Bureau of Mines, 1911, 23 pp.

[d] Katz, S. H., The absorption of methane and other gases by coal: Tech. Paper 147, Bureau of Mines, 1917, 22 pp.

[e] Taffanel, J., Le Floch, G., and Durr, A., Oxidation et distillation des poussières: Travaux de laboratoire, première série, Comité central des houillères de France, Station d'essais de Liévin, Paris, 1912, 67 pp.

[f] Taffanel, J., Le Flock, G., and Durr, A., Work cited.

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oxygen and the nitrogen content. The degree of accuracy of these calculations does not permit appreciating the magnitude of the loss of hydrogen and carbon referred to a determined quantity of fresh coal."

Mahler [a] heated 200 grams of powdered coal in a current of dry nitrogen to about 100° C. till no water was given off, as shown by the weight of a tube of sulphuric acid. The temperature was then lowered to 20° to 25° C. and dry air passed through the coal at a rate of a liter an hour for 30 hours. The water liberated was collected and weighed with the following results:

Water evolved from coal.

Coal.	Per cent volatile.	H_2O produced, grams per 200 grams of coal.
A.....	33	0.018
B.....	27	.00
C.....	25	.034
D.....	8	.00
E.....	2	.183

Here are trustworthy data which show that in low-temperature oxidation hydrogen is evolved from coal in the form of water, but the increase of water produced with the decrease of hydrogen in the coal seems anomalous.

Graham [b] passed a stream of dried air through desiccated coal at 30° C. and analyzed the coal before and after the experiment. He says:

> From the analyses of the moisture content of the coal-dust samples before and after "dry" oxidation, it is evident that quite a large amount of water is produced during oxidation at low temperatures. * * * It is not, however, possible to base any definite conclusion on these results, owing to the fact that anhydrous calcium chloride was placed in the oxidation bottles. * * * Since for the production of 1 gram of moisture, 620 c. c. of oxygen are necessary, it would seem that at low temperature oxidation (that is, below 50° C.) of dry-coal dust, a large amount of water is formed compared with the amount of oxygen absorbed.

Graham's method differed from that of Mahler in involving the heating of the coal after the oxidation, with the possible decomposition of the coal-oxygen complex formed and a consequent formation of water. However that may be, the presence of calcium chloride in the system next to the coal lessens the reliance to be placed on his work.

METHOD AND APPARATUS.

The apparatus used by the authors was similar to that used by Mahler and by Graham. From a drying train the gases passed

[a] Mahler, M. P., Expériences sur l'oxidation de la houille: Ann. des mines, t. 4, 1913, pp. 163-198.
[b] Graham, J. I., The absorption of oxygen by coal: Coll. guard, vol. 109, 1915, pp. 228-229.

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through a large quantity of coal at room temperature; thence they passed through another drying train which caught the water given up by the coal. This water was periodically weighed. The corresponding change of weight of the coal during the same time was also determined.

However, a different principle than that used by Mahler or by Graham was adapted by the authors.

The subcommittee on standardization of methods on determining water in coals[a] have shown that coal dried in a vacuum at room temperature loses water rapidly at first and then loses very small amounts for an extended period of time, when the curve for time vs. water lost by the coal becomes almost parallel to the time axis. Also, Porter and Ralston[b] have shown that as coal dries the relation of vapor pressure to water content is such that plotting the total water content against the total vapor pressure exerted by the coal produces a smooth even curve without sharp changes of direction.

The authors did not attempt to obtain an absolutely dry coal, which at best is an uncertain quality in view of the report of the committee mentioned, but dried the coal long enough to reach that part of the time-water curve parallel to the time axis. Thereafter the method employed was to observe vapor pressure of the coal through the weight of water removed by a regulated stream of dry gas. Nitrogen and air were alternately employed. A change in the direction of the drying curve would indicate a change in the vapor pressure due to formation of water by the oxidation of the coal.

DESCRIPTION OF APPARATUS.

Figure 1 shows the arrangement of the apparatus. The two large bottles on the left were arranged to form a gas holder with a capacity of 9 liters. One bottle was graduated for measuring the gas. The gas from the holder was dried by passing in succession through a calcium-chloride tower a wash bottle containing sulphuric acid and two U-tubes containing phosphorus pentoxide on glass wool. The ground-glass stoppers were arranged so that on being turned they closed the tubes. One of these stoppers was set to allow the gas to pass through at an average rate of a liter an hour. This setting was not disturbed during the entire experiment.

The coal container was made long and narrow, to insure the gas passing all parts of the coal. The mercury manometer and the small vesssel holding phosphorus pentoxide were used during the preliminary drying of the coal in a vacuum.

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[a] Lessing, R., Report of the subcommittee on standardization of methods on determining water in coals and other fuels and in minerals, of the international committee on analyses: Original communications, Eighth Intern. Cong. Appl. Chem., vol. 25, 1912, pp. 41–90.

[b] Porter, H. C., and Ralston, O. C., Some properties of the water in coal: Tech. Paper 113, Bureau of Mines, 1916, 28 pp.

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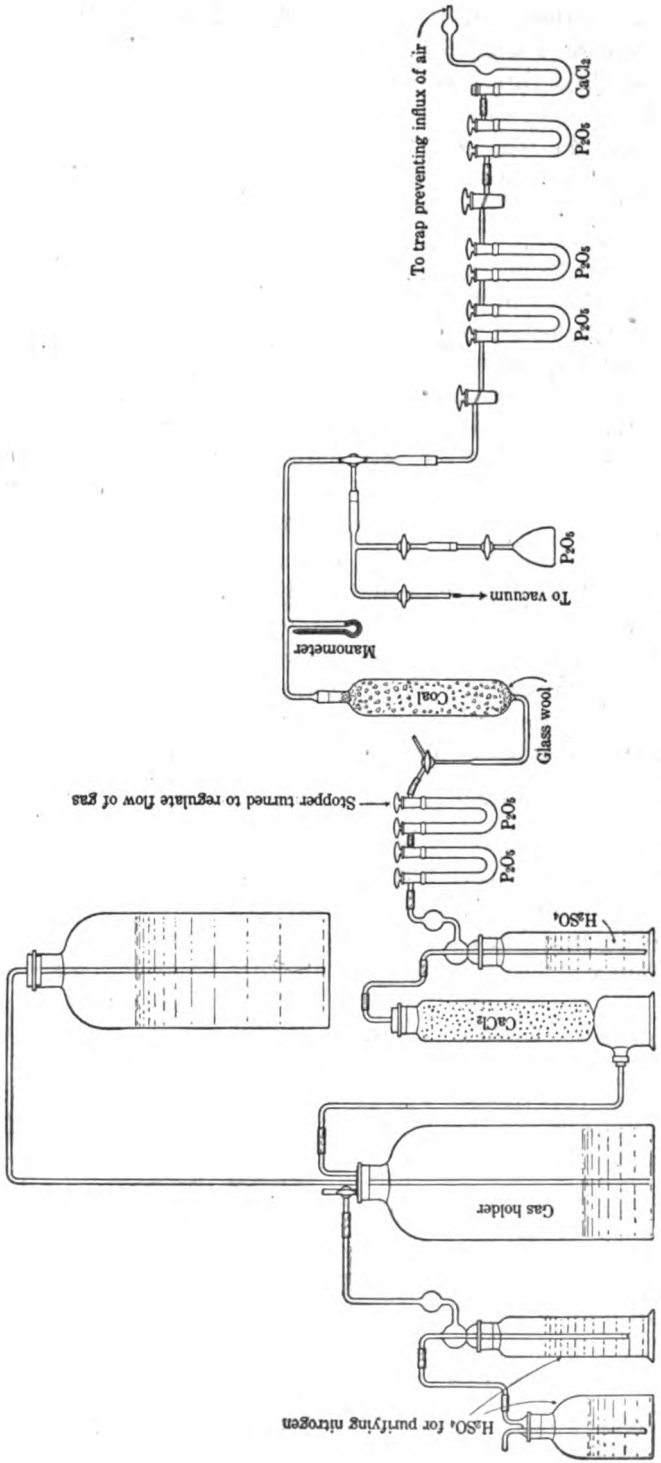

FIGURE 1.—Diagram of apparatus.

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The two U-tubes, following the coal container, held phosphorus pentoxide on glass wool for collecting the water removed from the coal. These two tubes, though shown as in a straight line, were placed side by side, so as to form a single compact piece for weighing. Beyond the weighing tubes were two guard tubes containing phosphorus pentoxide and calcium chloride.

COAL USED.

The coal was from the Pittsburgh seam of the Mansfield mine, at Carnegie, Pa., and had been mined only a few days before the experiment. Selected lumps were crushed to pass a 40-mesh screen; the coal that lodged on a 60-mesh screen was used for the experiment. A sample was taken for analysis. Crushing and screening were done as quickly as possible. The holder was filled with coal—about 270 grams. The air was immediately pumped out and the drying in a vacuum began.

GAS USED.

Nitrogen and air were used. The nitrogen was generated from commercial nitrogen, as sold in cylinders, by passing it from a cylinder through a Van Brunt nitrogen generator. Analyses of 10 different lots gave a maximum oxygen content of 0.25 per cent and a minimum of 0.05 per cent, the average being 0.13 per cent. Former experiments by the authors had shown that the amount of oxygen absorbed from this atmosphere by the coal would be negligible.[a]

PROCEDURE IN EXPERIMENT.

Much of the water in the coal was removed by drying in a vacuum. Then the coal was dried further by passing a stream of the dried nitrogen through it. When 550 liters of nitrogen had been used, the data showed that each liter of the gas was continuously removing about 1.6 mg. of water from the coal. Air was then passed through the coal, and the change in weight of the coal and the water evolved were recorded. The two gases were alternately used in this way throughout the experiment.

When the experiment was concluded, the coal was brought to equilibrium with the moisture in the air—a reverse air-drying process—and then analyzed.

ACCURACY OF THE GAS VOLUMES.

The volumes of gas used were corrected to atmospheric pressure and room temperature, any water vapor present being neglected. The small and constant error thus introduced does not affect the value of the results as a basis for conclusions. Otherwise the error of measurement was less than 2 per cent.

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a Katz, S. H., Absorption of methane and other gases by coal: Tech. Paper 147, Bureau of Mines, 1917, 22 pp.

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ACCURACY OF WEIGHT OF COAL.

The large surface of the coal container presented a source of error in weighing. Before each weighing the surface was carefully wiped with a clean cloth. The coal was put into the balance and allowed to stand half an hour before weighing. The total weight of coal and container was 500 grams. The balance had a capacity of one kilo, was sensitive to a milligram, and was glass inclosed. An accurate set of weights were used. Errors in weighing are undoubtedly within 10 mg.

ACCURACY OF WEIGHT OF WATER.

The U-tubes containing the water were weighed on a sensitive analytical balance. Before each weighing the glass was carefully

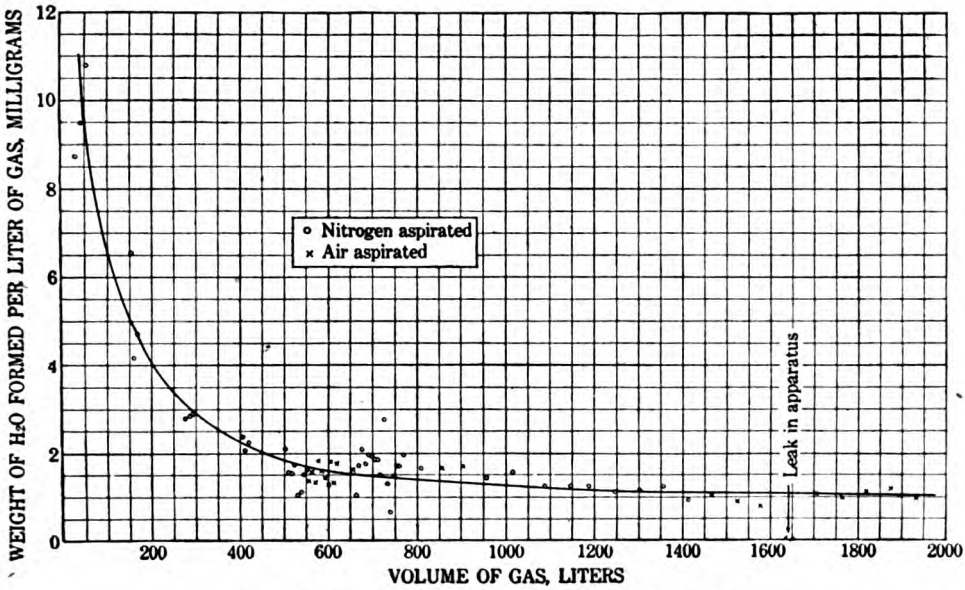

FIGURE 2.—Relation of moisture content of gas to volume of gas aspirated.

wiped with a clean cloth. The tubes were then allowed to stay for half an hour on the balance before weighing. The error was probably within a milligram.

RESULTS OF THE EXPERIMENT.

Table 1 presents the data of gas volumes and weights of water and coal obtained. The relations between volumes of gases and weight of water evolved by the coal as given in this table are shown graphically in figure 2.

Table 2 is a summary of the data in Table 1. Table 3 gives the analyses of the coal before and after the experiment.

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TABLE 1.—*Results of experiment showing water evolved and change in weight of coal.*

[Average rate of gas flow, 1 liter per hour.]

No. of test.	Kind of gas used.	Volume of gas at atmospheric conditions.		Coal.			Water evolved during test.		Remarks.
		Used in test.	Total used.	Weight.	Change of weight during test.		Total.	Per liter of gas.	
					Total.	Per liter of gas.			
		Liters.	*Liters.*	*Grams.*	*Mg.*	*Mg.*	*Mg.*	*Mg.*	
1	Nitrogen	27.9	27.9						
2	do	7.7	35.6				67.2	8.73	
3	do	8.3	43.9				78.7	9.48	
4	do	8.4	52.3				90.7	10.8	
5	do	92.9	145.2						
6	do	7.1	152.3				46.7	6.58	
7	do	7.1	159.4				29.6	4.17	
8	do	7.0	166.4				33.7	4.74	
9	do	111.5	277.9						
10	do	7.3	285.2				20.5	2.81	
11	do	7.2	292.4				20.6	2.86	
12	do	7.2	299.6				20.9	2.90	
13	do	97.7	397.3						
14	do	7.0	404.3				16.7	2.39	
15	do	7.0	411.3				14.5	2.07	
16	do	7.0	418.3				15.8	2.26	
17	do	77.0	495.3						
18	do	7.0	502.3				14.9	2.13	
19	do	7.0	509.3				11.0	1.57	
20	do	7.0	516.3				10.8	1.54	
21	do	7.0	523.3				13.7	1.71	
22	do	7.0	530.3				7.5	1.07	
23	do	7.0	537.3				7.8	1.11	
24	do	7.0	544.3				10.5	1.50	
25	do	7.0	551.3				11.4	1.63	
26	Air	7.0	558.3	263.347			9.6	1.36	
27	do	7.0	565.3				11.0	1.57	
28	do	7.0	572.3				9.4	1.34	
29	do	7.0	579.3				12.8	1.83	
30	do	7.0	586.3				11.2	1.60	
31	do	7.0	593.3				10.0	1.43	
32	do	7.0	600.3				9.1	1.30	
33	do	7.0	607.3	263.748	+401	+8.18	12.8	1.83	
34	do	7.0	614.3				9.3	1.33	
35	do	7.0	621.3				12.4	1.77	
36	do	35.0	656.3				57.1	1.63	
37	Nitrogen	7.0	663.3	263.887	+139	+2.48	7.4	1.06	
38	do	5.7	669.0				9.9	1.74	
39	do	7.0	676.0				14.7	2.10	
40	do	7.0	683.0				12.3	1.76	
41	do	7.0	690.0	262.789	(?)	(?)	13.7	1.96	Coal vessel was broken and mended; some coal lost.
42	do	7.0	697.0				13.6	1.94	
43	do	7.0	704.0				13.1	1.87	
44	do	7.0	711.0				13.1	1.87	
45	do	7.0	718.0				10.3	1.47	
46	do	7.0	725.0	262.784	− 5	−0.14	19.4	2.77	
47	do	7.0	732.0	262.759	− 25	−3.57	9.0	1.29	
48	do	7.0	739.0	262.720	− 39	−5.57	4.5	0.64	
49	do	7.0	746.0				10.3	1.47	
50	do	7.0	753.0				12.0	1.71	
51	do	7.0	760.0				11.9	1.70	
52	do	7.0	767.0	262.679	− 41	−1.46	13.8	1.97	
53	do	42.0	809.0	262.664	− 15	−0.36	69.7	1.66	
54	Air	49.0	858.0	262.798	+134	+2.73	82.0	1.67	
55	do	49.0	907.0	262.914	+116	+2.37	84.5	1.72	
56	Nitrogen	49.3	956.3	262.818	− 96	−1.95	70.3	1.43	
57	do	56.3	1,012.6	262.802	− 16	−0.28	89.0	1.58	
58	do	42.2	1,054.8	262.739	− 63	−1.49			
59	do	35.1	1,089.9	262.700	− 39	−1.11	45.1	1.29	
60	do	56.4	1,146.3	262.628	− 72	−1.28	70.8	1.26	
61	do	42.2	1,188.5	262.587	− 41	−0.97	52.8	1.25	
62	do	56.4	1,244.9	262.530	− 57	−1.01	63.3	1.13	
63	do	56.4	1,301.3	262.401	−129	−2.29	65.0	1.15	
64	do	56.1	1,357.4	262.430	+ 29	+0.52	70.2	1.25	
65	do	54.6	1,412.0	262.391	− 39	−0.71	50.8	0.93	

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TABLE 1.—*Results of experiment showing water evolved and change in weight of coal*—Continued.

No. of test.	Kind of gas used.	Volume of gas at atmospheric conditions.		Coal.			Water evolved during test.		Remarks.
		Used in test.	Total used.	Weight.	Change of weight during test.		Total.	Per liter of gas.	
					Total.	Per liter of gas.			
		Liters.	*Liters.*	*Grams.*	*Mg.*	*Mg.*	*Mg.*	*Mg.*	
66	Air	56.5	1,468.5	262.506	+115	+2.04	61.0	1.08	
67	...do	56.1	1,524.6	262.570	+ 64	+1.14	50.5	.90	
68	...do	54.0	1,578.6	262.627	+ 57	+1.06	42.6	.79	
69	...do	57.1	1,635.7	262.677	+ 50	+ .87	3.7	.06	Leak in apparatus.
70	...do	14.2	1,649.9	262.729	+ 52	+3.66	0.2	.01	Leak stopped.
71	...do	55.3	1,705.2	262.779	+ 50	+ .90	60.2	1.09	
72	...do	57.1	1,762.3	262.811	+ 32	+ .56	56.4	.99	
73	...do	56.8	1,819.1	262.857	+ 46	+ .81	64.6	1.14	
74	...do	56.7	1,875.8	262.946	+ 89	+1.57	67.6	1.19	
75	...do	56.7	1,932.5	262.967	+ 21	+ .37	56.3	.99	

TABLE 2.—*Summary of data in Table 1.*

No. of tests.	Kind of gas used.	Volume of gas at atmospheric conditions.		Coal.			Water evolved during test.		Remarks.
		Used in test.	Total used.	Weight.	Change in weight during test.		Total.	Per liter gas.	
					Total.	Per liter gas.			
		Liters.	*Liters.*	*Grams.*	*Mg.*	*Mg.*	*Mg.*	*Mg.*	
1 to 17	Nitrogen	495.3	495.3	.....	.....	.....	.....	.....	
18 to 25	...do	56.0	551.3	263.347	.....	.....	87.6	1.56	
26 to 36	Air	105.0	656.3	263.887	+540	+5.14	164.7	1.57	
37 to 41	Nitrogen	33.7	690.0	262.789	(?)	(?)	58.0	1.72	Some coal lost through accident.
42 to 53	...do	119.0	809.0	262.664	−125	−1.05	200.7	1.69	
54 and 55	Air	98.0	907.0	262.914	+250	+2.55	166.5	1.70	
56 to 65	Nitrogen	505.0	1,412.0	262.391	−523	−1.04	577.3	1.25	Test 58 omitted in figuring H_2O per liter of gas.
66 to 75	Air	449.2	1,861.2	262.865	+474	+1.06	459.2	1.02	Tests 69 and 70 omitted in calculations.

TABLE 3.—*Analyses of the coal used.*

Item.	Before experiment.		After experiment.	
	Coal, air dried.	Coal, moisture and ash free.	Coal, air dried.	Coal, moisture and ash free.
Air-drying loss, per cent	0.5	.....	a 1.1	.....
Proximate analysis:				
Moisture, per cent	1.55	.....	1.94	.....
Volatile matter, do	37.00	40.00	36.36	39.19
Fixed carbon, do	55.51	60.00	56.43	60.81
Ash, do	5.94	.....	5.27	.....
Total	100.00	100.00	100.00	100.00

a Gain.

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TABLE 3.—*Analyses of the coal used*—Continued.

Item.	Before experiment.		After experiment.	
	Coal, air dried.	Coal, moisture and ash free.	Coal, air dried.	Coal, moisture and ash free.
Ultimate analysis:				
Hydrogen.....per cent..	5.35	5.61	5.34	5.53
Carbon.....do....	77.00	83.23	76.91	82.90
Nitrogen.....do....	1.58	1.71	1.54	1.66
Oxygen.....do....	8.96	8.18	9.79	8.67
Sulphur.....do....	1.17	1.27	1.15	1.24
Ash.....do....	5.94	.....	5.27	.....
Total.....	100.00	100.00	100.00	100.00
Calorific value:				
Determined.....	7,703	8,326	7,651	8,246
Calculated from ultimate analysis.....	7,706	8,335	7,660	8,260

DISCUSSION OF THE RESULTS.

When nitrogen was passed through the coal there was invariably a loss in weight. The loss, however, was much less than the weight of water evolved from the coal. Hillebrand and Badger[a] found a similar discrepancy which they could not explain. The small amount of oxygen in the nitrogen used by the authors is much insufficient to account for the discrepancy observed. They suggest as a possible explanation that it may be due to absorption of nitrogen by the coal which has been shown to occur.[b]

When air was passed through the coal there was always a gain in weight despite the loss of water.

Comparison of the analyses, on a moisture and ash-free basis, of the coal before and after the experiments shows the increased proportion of oxygen and the corresponding decrease in other constituents. No loss of hydrogen from the coal substance can be shown by the analyses.

The vapor-pressure curve, as shown by the graph, figure 2, follows the same regular course regardless of whether air or nitrogen was passed over the coal, and the coal became gradually drier throughout the experiment. No evidence is afforded that water is produced by the oxidation of coal at ordinary temperature.

CONCLUSIONS.

1. There is a lack of agreement between the weight of water evolved by coal and the loss of weight when dried in an inert atmosphere. The excess weight of the coal may be due to absorption of gas.

2. A study of the vapor tension of water in coal, as indicated by the water removed by a regulated current of dry nitrogen and air used alternately, shows no production of water by the oxidation of coal at ordinary temperatures.

[a] Hillebrand, W. F., and Badger, W. L., Errors in the determination of moisture in coal: Original communications, Eighth Intern. Cong. Appl. Chem., vol. 10, 1912, p. 187.

[b] Katz, S. H., Absorption of methane and other gases by coal: Tech. Paper 147, Bureau of Mines, 1917, 22 pp.

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